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CHEMICAL PRODUCTS DIVISION

RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

A REPORT TO

OFFICE OF ADVANCED RESEARCH AND
TECHNOLOGY, NATIONAL AERONAUTICS
AND SPACE ADMINISTRATION
WASHINGTON, D.C.

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AEROJET-GENERAL CORPORATION

AZUSA, CALIFORNIA

Chemical Products Div.

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RESEARCH ON PROCESSES
FOR UTILIZATION OF LUNAR RESOURCES

Quarterly Report
[22 Apr. - 31 July 1963]

a report to

OFFICE OF ADVANCED RESEARCH AND TECHNOLOGY
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C.

S.D. Rosenberg, G. A. Guter, and F. E. Miller

(NASA Contract NAS 7-225)

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(NASA CR-52318)

Report
Report No. 0765-01-1 (Quarterly)

August 1963 *29p*

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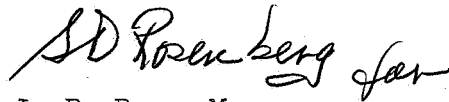
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AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

This report is submitted in partial fulfillment of Contract NAS 7-225.

The period covered by the report is 22 April 1963 through 31 July 1963.

AEROJET-GENERAL CORPORATION

A handwritten signature in dark ink, appearing to read "L. R. Rapp", followed by a stylized flourish or "for" written in cursive.

L. R. Rapp, Manager
Chemical Products Division

ABSTRACT

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Laboratory apparatus for studying the catalytic reduction of carbon monoxide with hydrogen has been designed, fabricated, and operated. Excellent yields of methane (to 99.0%) and water (to 100%) were achieved; minor amounts of carbon dioxide were formed as a by-product. A negligible amount of carbon (less than 0.1%) was deposited on the catalyst during several hours of operation. The reaction appears very promising as a step in the reduction of silicate materials (lunar raw materials) to produce oxygen.

AUTHOR

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I. OBJECTIVE

The objective of this program is to study the reaction of carbon monoxide and hydrogen, under a variety of conditions, to yield water and a carbon-containing material, preferably methane. Sufficient data will be obtained to permit a preliminary evaluation of this reaction as a step in the reduction of silicate materials (lunar raw material) to produce oxygen.

II. SUMMARY

Research under Contract NAS 7-225 was initiated on 22 April 1963. The experimental work is to be completed on 15 November 1963. The program is divided into six major tasks: Task 1, Design; Task 2, Fabrication; Task 3, Testing; Task 4, Data Acquisition; Task 5, Process Integration; and Task 6, Reports.

Under Tasks 1, 2 and 3, a flow reaction tube was designed along the lines of a Fischer-Tropsch reactor. The tube was fabricated and successfully tested with argon at 5 atmospheres and 1000°C. The apparatus functioned smoothly in three test runs with carbon monoxide and hydrogen.

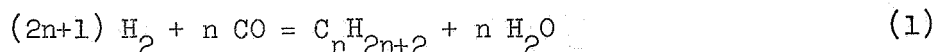
Under Task 4, highly encouraging results were obtained in the initial experiments involving the reduction of carbon monoxide with hydrogen to form methane and water. The carbon monoxide conversions were excellent (98.6 to 99.4%). Yields of methane (88.4 to 99.0%) and water (76.5 to 100%) were extremely good. Yields of by-product carbon dioxide were low (1.7 to 10.9%). Less than 0.1% of the total carbon passed through the reactor was deposited on the catalyst bed - a negligible loss of material.

The program is proceeding on schedule. Future work will continue investigations of reaction variables such as pressure, reactant ratio, catalyst, and catalyst poisoning.

III. TECHNICAL DISCUSSION

A. LITERATURE REVIEW

The literature on the reaction of carbon monoxide with hydrogen has been studied with care during this report period. Due to the copious amount of work which has been done on the many facets of this reaction and the rapid pace with which the program is required to move, it has been necessary to rely heavily on previous literature surveys. Two literature reviews (References 1 and 2) were found to be of particular value as aids in locating the most recent pertinent work. Reference 1 contains a wealth of thermodynamic data for the reactions



and several related reactions producing a variety of other products.

Reference 2 is a bibliography containing 3711 abstracts of papers and 4017 abstracts of patents. These sources have been helpful in locating the most recent literature (References 3 through 7) on the reaction to form methane as a primary product. Investigation 5137 of the U.S. Bureau of Mines was very helpful in the design of the equipment and in the choice of the catalysts. This report appeared to be the most recent of significant work on the reaction and is representative of the state of the art before the present program was initiated.

Although much work was devoted to studies on the production of methane, such work was directed toward objectives which are considerably different from the objectives of this program. Previous investigators sought a hydrocarbon product of commercial interest and had little regard for water production or precise material balances. Because this investigation is concerned with a reaction intended for use on the lunar surface, water now becomes an important product, and loss of carbon, hydrogen, and oxygen in the reaction is a critical factor to be considered. These broad objectives were borne in mind when the literature was reviewed to select data for design, construction, and operating conditions of the flow apparatus.

B. TASK 1, DESIGN

The equipment to be used in the study of the reaction of carbon monoxide with hydrogen was designed to allow maximum flexibility in operating conditions. Type 316 stainless steel was chosen as the reaction-chamber metal because of its high temperature strength and resistance to corrosion. The U.S. Bureau of Mines (Reference 3) used stainless steel chambers successfully in its laboratory scale equipment.

Figure 1 is a schematic flow diagram of the hydrogen-carbon monoxide reactor designed for this investigation. Heat dissipation is one of the major problems associated with the reaction as it is highly exothermic. An attempt was made to minimize this problem by making the reaction chamber (1)* small in diameter (0.527 in. ID) in relation to its length (38.5 in.). This design provides a large surface area for cooling and a minimum distance for the reacting gases to travel from the center of the chamber to the cooling surface. Fins are provided on the outside of the tube to furnish additional cooling surface. For convenience, air is used as the cooling fluid. In a lunar installation, a fluid such as Dowtherm or a molten salt, would be recirculated through an exchanger or radiator to dissipate the heat of reaction.

The reaction chamber is divided into three separate sections for temperature control. Each section is provided with an electrical heating unit (2) and a separate air-cooling system. This provides the unit with capability for adjusting the temperature profile of the reactor as desired.

Commercial grade hydrogen and carbon monoxide from compressed gas cylinders require two-stage pressure-reducing regulators, needle valves and rotameters to control the flow rates. The pressure on the reactor is controlled by a motor valve (11) which is operated by an automatic pressure recorder and controller (not shown).

Electric heaters (3) are provided for each of the two feed-gas streams for close temperature control. A small gas compressor (7), a rotameter (5), and a heater (3) are provided for the gas recycle stream. The outlet gas is cooled and passed through the condenser (8). The condensate is separated and collected in a receiver (9) and then measured (10). Samples of the outlet gas are collected for analysis (12) and the outlet gas volume is measured with a wet test meter (13).

* Numbers refer to coding system used in Figure 1 to label reactor components.

Chromel-Alumel thermocouples (4) are used to measure the catalyst bed temperatures. Skin thermocouples (thermocouples welded to the outside of the tube) are used to measure the intermediate bed temperatures. Inlet, outlet, and two midpoint bed temperatures are sensed by thermocouples immersed in the gas stream. The temperatures are measured and recorded by a 24-point strip chart recorder.

Sample connections have been provided at the $1/3$ and $2/3$ intermediate points in the catalyst bed. These connections consist of $1/4$ -in. stainless steel tubes welded to the chamber wall.

Task 1 was completed on 31 May 1963.

C. TASK 2, FABRICATION

The catalyst tube (bed) was fabricated from a 4-ft piece of $5/8$ -in. OD, 0.049-in. wall 316 stainless steel tube. Three 1-ft sections of stainless steel (serrated, $1-1/4$ in. OD, 4 fins/ft) fins were attached to the outside of the tube. Figure 2 is a photograph of the reaction unit under construction. In the photograph, the lower heating unit is being installed at the bottom of the tube. The lower air inlet connection was installed as shown. It is partly covered by insulation. The cooling air enters at the bottom, flows up around the stainless steel fins, and leaves at the top of each heating unit.

The carbon monoxide, hydrogen and recycle gas preheaters can be seen at the top of the photograph. Electric heaters similar to the bed heaters were later installed on the preheaters. The product gas cooler-condenser (lower right of photograph) is constructed of a 3-ft-long coil of $3/8$ -in. stainless steel tubing in a 4-in. OD stainless steel jacket. The condensate separators are made from standard 125-ml sample containers.

Figure 3 is a photograph of the completed reaction unit. The reactor and preheaters have been covered with a 3-in. layer of insulation and a stainless steel jacket. A wet test meter (lower right) was added to calibrate the reactant rotameters (not shown) and to measure the product gas. Graduated cylinders (100 ml) are used to receive the condensate. A panel board (left side of photograph) was

installed to mount the major instrumentation, including reactor bed pressure indicator-controller, reactor bed pressure drop recorder, temperature recorder (24-point strip chart), reactor catalyst bed temperature controllers, reactant gas preheater controllers, and pressure gages. Two-stage pressure-reducing regulators were mounted on the gas cylinders (not shown). The two reactant gas rotameters are mounted on the bench rack back of the panel board. The three cooling air rotameters and needle valves are also located on the bench rack to the left of the reactor (behind the panel board).

Task 2 was completed on 28 June 1963.

D. TASK 3, TESTING

1. Equipment Operational Limits

A series of short tests was made on the equipment in order to determine its operating limits and characteristics. Argon was used through both preheaters and through the reactor (no catalyst) to establish that reaction temperatures as high as 900°C can be obtained. The temperature on each of the three catalyst bed sections can easily be controlled independently to within as little as $\pm 2^{\circ}\text{C}$ or to as much as $\pm 200^{\circ}\text{C}$ of each other.

The catalyst bed pressure-control system was found to operate satisfactorily at pressures up to about 90 psig. With minor changes the equipment can be made to function from 7 psia up to 315 psia. Reactant flow rates can be accurately measured and controlled at rates as low as 1 scfh and as high as 20 scfh.

2. Catalyst Reduction

After the apparatus was tested for gas leaks at 200 psig, the reactor tube was filled with 65.4 g (139 cc) of catalyst C-0765-1005 (25% NiO on silica gel, 4 to 8 mesh). The bed temperature was maintained between 350 and 414°C with 1.0 atmosphere of hydrogen passing over the catalyst. These conditions were maintained for 5.0 hours; a sample of gas was removed every hour. Mass spectrographic analyses (completed in 0.5 hour after the sample was taken) revealed that the water content of the exit gas dropped to 0.3% from a maximum of 0.8%. Hydrogen pressure (60 psig) was maintained in the apparatus on shutdown.

3. Preliminary Runs with Carbon Monoxide and Hydrogen

Several highly successful preliminary test runs were made. These data are presented in Tables 1, 2, and 3, along with data from later runs. These preliminary runs were made with a 4 to 1 hydrogen/carbon monoxide mole ratio (2 scfh of hydrogen and 0.5 scfh of carbon monoxide) at 1.0 atmosphere pressure. The temperature profiles in the catalyst chamber were irregular but the conversions and yields were good. The overall material balances were excellent (to $\pm 1\%$). The individual component balances, however, were not so good, indicating that the operating and analytical techniques needed to be improved.

The operating data for the three preliminary runs are presented in Table 1; Table 2 presents the product gas analyses (mass spectrometer). The hydrogen concentration in the product gas varied from about 63 to 70% and the methane concentration from about 25 to 33%. Ideally, with a 4 to 1 hydrogen/carbon monoxide mole ratio, the composition should be 50% methane and 50% hydrogen. The carbon monoxide concentration varied from about 0.5 to 0.2%, corresponding to carbon monoxide conversion of 98.6 to 99.4%. The carbon dioxide concentration varied from about 5% to 1%. Only inconclusive traces of C_2 or heavier hydrocarbons were found in the product gas. The water which was condensed from the product gas was clear and colorless; vapor-phase gas chromatographic analysis showed that no carbon-containing compounds were present.

Table 3 presents the material balances for the runs. Carbon monoxide conversions were extremely good (98.6 to 99.4%). Yields of methane were fairly good (81.7 to 94.1%). Yields of water were good (85.5 to 90.1%) and yields of carbon dioxide were gratifyingly low (3.2 to 10.8%), compared with 25 or 30% reported in the literature. Run 5, with low temperatures in the top section and high temperatures in the bottom section of the bed, was found to give better yields of methane and water than Runs 8 and 9, in which the temperatures were all at 300°C or lower.

Task 3 was completed on 1 July 1963.

E. TASK 4, DATA ACQUISITION

A series of 11 successful data-producing runs was made (Runs 10 to 13, and 18 to 24) this quarter with the C-0765-1005 catalyst. The data for these runs are presented in Tables 1 to 4 and Figures 4 to 6. The first batch of catalyst was removed after Runs 14 and 15 showed that its activity had been somewhat reduced by operating at the high temperature (500°C).

1. Temperature vs Conversion and Yields

Figure 4 is a plot of the data showing how catalyst temperature affects carbon monoxide conversion and yields at a 4 to 1 hydrogen/carbon monoxide mole ratio at atmospheric pressure and a 500 hr^{-1} space velocity. In this plot the normalized yields were used (see Table 3). Although rather good material balances were obtained ($\pm 5\%$), the rather small changes in water and methane yields were sometimes partly obscured by the difference in material balance. Because the material balances vary somewhat above and below 100% and because the yields are directly affected by material balance, it is believed that it is proper to normalize the yields to 100% elemental balance.

Three different methods may be used to calculate product yields. The per pass yield in the case of methane is $(\text{moles of CH}_4 / \text{moles of CO})(100)$; the actual yield in this case is $(\text{moles of CH}_4 / \text{moles of CO} - \text{moles of CO unconverted})(100)$. Due to the inherent errors in operating the apparatus with continuously metered inputs and outputs, and with only intermittent sampling of gases for analysis, it is possible that a yield calculated by the above two methods may exceed 100%. Because all of the product appeared either in the gaseous phase or as condensed water, as indicated by the analysis of the catalyst bed after operation, the valid assumption was made that calculated material balances for the elements were in error by the amount they deviated from 100%. Consequently, the actual yield of any product was converted to a more significant yield by making use of this assumption.

An example of this conversion will now be given for Run 13 (Table 3). The actual yield of methane was calculated to be 98.0%. However, the material balance for carbon was calculated to be 104% because the total carbon in the products was found to be 7.469 g per hour whereas the input rate was only

7.17 g of carbon per hour. The methane carbon amounted to 6.954 g of the 7.469 g of total carbon. The amount of carbon in each of the products must be divided by 1.04 to bring the total carbon in the product down to equal 7.17 g. Consequently, the methane actual yield must also be reduced by this factor. The normalized yield is thus calculated as (actual yield/% material balance of major element)(100).

The oxygen balance was used to normalize the water yield because most of the oxygen in the reaction products was contained in the water; the carbon balance was used to normalize the methane yield because nearly all of the carbon in the reaction products was contained in the methane. Although the differences between the actual yields and normalized yields were small, the normalized yields gave a smoother plot of the data than did the actual yields. Also, the normalized yields approximated more closely the methane and water relationship which is expected when only methane, water, and carbon dioxide are formed.

A temperature of 400 to 450°C appears to be optimum for the 4 to 1 hydrogen/carbon monoxide ratio, yielding the maximum quantity of water and methane and the minimum quantity of carbon dioxide. At low temperatures, carbon dioxide is thermodynamically favored over carbon monoxide in the water-gas reaction (Reference 1).



It appears from the data that the rate of hydrogenation of carbon dioxide increases sufficiently with temperature to almost completely eliminate it in the product at 400 to 450°C. At temperatures above this range, competition between the rate of removal of carbon dioxide by hydrogenation and the rate of production of carbon dioxide by the water gas reaction begins to favor carbon dioxide production. This change in relative rates can most logically be attributed to a change in catalyst activity as the catalyst bed temperature is increased.

The second charge of catalyst (dotted lines in Figure 4) appeared to be more active than the first charge which was heated to higher temperatures in the preliminary runs. Excellent yields of 98 to 99% water and methane, and less than 1% carbon dioxide, were found to be obtainable with active catalyst.

2. Hydrogen/Carbon Monoxide Ratio

Figure 5 shows the effect of lowering the hydrogen/carbon monoxide ratio on conversion and yields. Lowering the hydrogen/carbon monoxide ratio from 4 to 1, to 3.1 to 1 (at 500 hr⁻¹ space velocity and 450°C) lowered the water and methane yields severely (95.9% to 78.5%, and 98.1% to 88.4%, respectively) while increasing the carbon dioxide yield 5-fold (2.1 to 10.9%). At the same time the methane content of the product gas increased from 48.9% to 56.8% (see Table 2) - a relatively small change. More work must be done to substantiate these results and to determine if lower space velocities or higher pressures or higher catalyst activity will improve the yields under these more nearly stoichiometric conditions.

3. Space Velocity

Space velocity, as used in this report, is defined as the reactant flow rate (scfh) divided by catalyst volume (cu ft). Increasing the space velocity from 500 to 1000 hr⁻¹ (at 4 to 1 hydrogen/carbon monoxide mole ratio and 450°C) was found (see Figure 6) to decrease water and methane yield appreciably (95.9% to 89.4%, and 98.1% to 90.3%, respectively) while drastically increasing the carbon dioxide yield (2.1% to 9.2%). The methane content of the product gas also fell (from 48.9% to 33.7%). It appears that lower space velocities should be investigated, especially at lower hydrogen/carbon monoxide ratios. Higher space velocities may also be used at higher pressures.

4. Pressure

Only atmospheric pressure has been used thus far. Higher pressures will be investigated.

5. Catalysts

Only one catalyst has been evaluated to date. Other catalysts which may be active at lower temperatures will be tested.

6. Recycle

Originally, recycle runs were anticipated. However, because such excellent conversions and yields have been obtained with the simple single pass operation, no recycle runs are now contemplated.

7. Carbon Deposition

Carbon formation or deposition does not appear to be a problem under the conditions tested to date. The first charge of catalyst was removed after about 56 hours of operation. Analysis of this catalyst showed that only about 2 wt% of carbon was deposited in the top 1/3 of the bed. No carbon was deposited on the lower 2/3 of the catalyst. The carbon deposited on the top 1/3 of the bed represented less than 0.1% of the carbon charged over the 56-hour operating time.

8. Higher Hydrocarbons

No evidence was found (see Table 2) of higher hydrocarbons in the product gas or in the water condensed from the runs at atmospheric pressure.

9. Catalyst Bed Depth

These runs were made with a relatively deep catalyst bed (39 in.) as compared to 4 in. to 12 in. for most of the investigations found in the literature. This may, in part, be the reason that why such high conversions to desired products were obtained in the work reported herein. Of particular note is the reconversion of carbon dioxide into methane and water.

Table 4 presents representative gas analyses of samples taken from the intermediate catalyst bed sample points. This data shows that although most of the carbon monoxide was converted in the top section of the bed, the lower two feet of the bed were required to convert the carbon dioxide formed in the top foot of the bed into methane and water. This ability to convert carbon dioxide to required product is most important. Every effort will be made to eliminate this undesired by-product from the product gas.

F. TASK 5, PROCESS INTEGRATION

The data obtained in Task 4 will be used to integrate the carbon monoxide reduction reaction into the overall carbothermal process for the preparation of oxygen by the reduction of silicate rock. No work on this task was accomplished during the present report period. Such work will be accomplished during the last month of the program.

G. TASK 6, REPORTS

The first two monthly reports were submitted in June and July. The remaining two monthly reports will be submitted in September and October. The second quarterly report will be submitted in November 1963 and the final report in December 1963.

IV FUTURE WORK

A. TASK 4, DATA ACQUISITION

1. Space Velocity

Several more points will be obtained to confirm and extend the space velocity relationships.

2. Pressure

A series of points will be run at higher pressures (up to 90 psig on the catalyst bed).

3. Hydrogen/Carbon Monoxide Ratio

Several points will be obtained at lower hydrogen/carbon monoxide mole ratios at low space velocities and/or at high pressures in an attempt to increase the concentration of methane in the product gas.

4. Catalysts

One or more catalysts will be tested in an attempt to find one which will be more active at the lower temperatures which favor the equilibrium for the conversion of carbon dioxide into methane and water.

5. Catalyst Poisoning

A series of tests will be made in order to determine the effect of various impurities in the feed upon catalyst life or activity. Impurities to be tested include sulfur compounds, carbon dioxide, and oxides of phosphorus and nitrogen.

B. TASK 6, REPORTS

Reports will be submitted as scheduled.

V. PERSONNEL

The senior staff assigned to this program was comprised of S. D. Rosenberg (Project Engineer), G. A. Guter, F. E. Miller, and G. R. Jameson.

VI. EXPENDITURES AND COMMITMENTS

Approximately 1665 manhours and \$23,000 were expended on Contract NAS 7-225 during this report period.

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TABLE 1
REDUCTION OF CARBON MONOXIDE WITH HYDROGEN
(OPERATING DATA)^a

Run No.	Reactant Flow Rate		Product Flow Rate		Inlet Gas Temp (°C)	Catalyst Bed Temperature			Outlet Gas Temp (°C)
	H ₂ (scfh)	CO (scfh)	Gas (scfh)	H ₂ O(l) (g/hr)		Top (°C)	Mid (°C)	Bottom (°C)	
5 ^b	2.02	0.48	1.39	9.1	220	178	311	541	228
8	2.00	0.50	1.57	8.8	281	280	275	278	170
9	2.00	0.50	1.28	8.2	240	259	300	299	169
10	2.00	0.50	1.207	8.66	290	348	350	350	195
11	2.00	0.50	1.008	9.64	314	382	399	401	219
12	2.00	0.50	1.056	9.97	291	448	451	451	248
13	2.00	0.50	1.086	9.38	321	500	500	501	283
18	2.00	0.50	0.990	10.32	269	350	351	351	182
19	2.00	0.50	1.022	10.04	307	402	403	402	204
20	2.00	0.50	1.009	10.05	320	452	451	452	251
21	2.12	0.69	1.069	11.45	232	450	451	452	239
22	2.88	0.88	1.553	13.76	326	450	453	452	266
23	2.88	0.88	1.540	13.40	328	452	453	451	261
24	3.90	0.96	2.461	14.72	277	454	452	452	264

^aCatalyst: C-0765-01-1005; Bed Pressure: 1 atmosphere.

^bRuns 5, 8 and 9 were preliminary runs used to test the operational characteristics of the apparatus.

TABLE 2REDUCTION OF CARBON MONOXIDE WITH HYDROGEN
(PRODUCT GAS ANALYSIS)^a

Run No.	Composition of Product Gas (vol%)				
	H ₂	H ₂ O	CO	CH ₄	CO ₂
5	65.4	1.20	0.50	32.0	0.9
8	70.0	1.20	0.30	25.8	2.7
9	63.5	1.20	0.20	30.9	4.2
10	58.97	1.21	0.38	35.84	3.59
11	50.47	1.21	0.34	46.86	1.13
12	52.16	1.21	0.39	45.42	0.82
13	50.95	1.21	0.52	44.54	2.78
18	45.80	1.21	0.33	51.67	0.99
19	51.68	1.21	0.24	46.54	0.33
20	48.51	1.21	0.30	48.96	1.02
21	34.55	1.21	0.50	56.82	6.93
22	41.45	1.21	0.52	50.81	6.01
23	43.36	1.21	0.59	48.85	5.99
24	60.91	1.21	0.69	33.70	3.49

^aThree to six gas samples were obtained during the course of each run, at 1- to 2-hour intervals. The average analysis resulting from these samples for each run is reported here. The composition of the gas was determined by mass spectrometric analysis, excepting water vapor. The water vapor concentration was assumed to be fixed by the condenser water temperature (10°C). This assumption was spot-checked periodically by vapor-phase chromatography and was found to be correct. Each gas sample was checked for higher hydrocarbons; none were found. Liquid water samples were spot-checked for carbon-containing compounds; none were found.

TABLE 3
REDUCTION OF CARBON MONOXIDE WITH HYDROGEN
(CO CONVERSION AND PRODUCT YIELD)

Run No.	H ₂ /CO Mole Ratio	Space Velocity ^a (hr ⁻¹)	Nominal Catalyst Bed Temp ^b (°C)	Overall Material Balance (%)	CO Conversion (mole%)	Actual Product Yield (mole%)			Normalized Product Yield ^d (mole%)		
						H ₂ O ^c	CH ₄	CO ₂	H ₂ O ^c	CH ₄	CO ₂
5	4.2	503	-	100	98.6	90.1	94.1	3.2			
8	4.0	503	278	101	99.4	85.5	81.7	8.5			
9	4.0	503	285	101	99.4	88.0	79.4	10.8			
10	4.0	503	350	99.6	99.1	84.2	87.4	8.7	83.1	90.5	8.7
11	4.0	503	394	97.2	99.3	92.6	95.4	2.3	95.2	97.7	2.3
12	4.0	503	450	99.3	99.1	96.1	96.9	1.7	96.6	98.1	1.7
13	4.0	503	500	102.5	89.9	90.8	98.0	6.1	88.3	94.3	6.1
18	4.0	516	351	102.8	99.3	98.9	103.2	2.0	96.2	98.4	2.0
19	4.0	516	402	97.4	99.5	98.0	95.6	0.7	100.1	99.0	0.7
20	4.0	516	452	100.5	99.4	96.5	99.5	2.1	95.9	98.1	2.1
21	3.1	580	451	100.8	99.2	79.8	89.1	10.9	78.5	88.4	10.9
22	3.3	778	452	98.4	99.1	74.8	89.9	10.6	76.9	89.6	10.6
23	3.3	778	452	95.6	99.0	73.1	85.8	10.5	76.5	88.7	10.5
24	4.1	1005	453	96.1	98.2	75.5	89.8	9.1	79.2	90.2	9.1

^aSpace Velocity = $\frac{\text{Standard cu ft/hr of reactants}}{\text{cu ft of catalyst}}$

^bAverage of the top, mid, and bottom temperature points of the catalyst bed.

^cBased on total of liquid and gaseous fractions.

^dBased on 100% material balance for oxygen (calculated as water) and for carbon (calculated as methane and carbon dioxide).

TABLE 4
REACTION GAS COMPOSITION^a

Run No.	Sample Location	Gas Composition (vol%)			
		H ₂	CO	CH ₄	CO ₂
10-4a	Top	70.6	0.4	18.8	4.9
10-4b	Mid	61.1	0.4	34.1	3.9
10-4c	Outlet	58.2	0.3	37.3	3.6
11-6a	Top	-	-	-	6.8
11-6b	Mid	-	-	-	2.4
11-6c	Outlet	54.5	0.3	43.5	1.1
19-3a	Top	-	-	-	6.0
19-3b	Mid	-	-	-	1.3
19-3c	Outlet	54.1	0.1	45.1	0.3

^aGas samples were removed from the reactor, 12 and 24 in. from the top of the catalyst bed, respectively, and analyzed. The results are compared with the product gas analysis.

1 Stainless Steel Flintube, Reaction Chamber

2 Heaters

3 Heaters

4 Thermocouples

5 Flow Indicators

6 Pressure Reducing Regulators

7 Gas Recycle Compressor

8 Cooler-Condenser

9 Condensate Receiver

10 Graduate Cylinder

11 Back Pressure Regulator

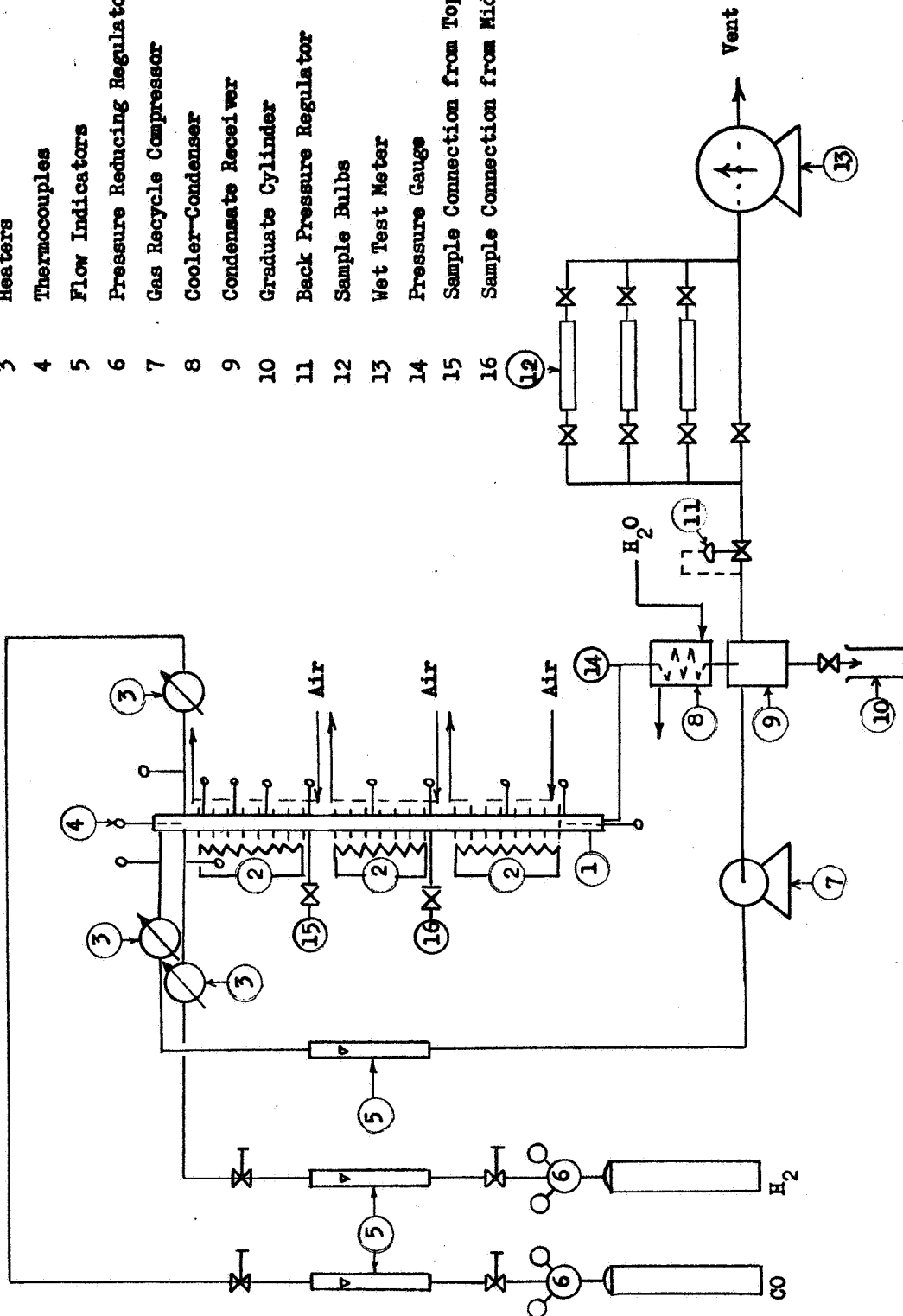
12 Sample Bulbs

13 Wet Test Meter

14 Pressure Gauge

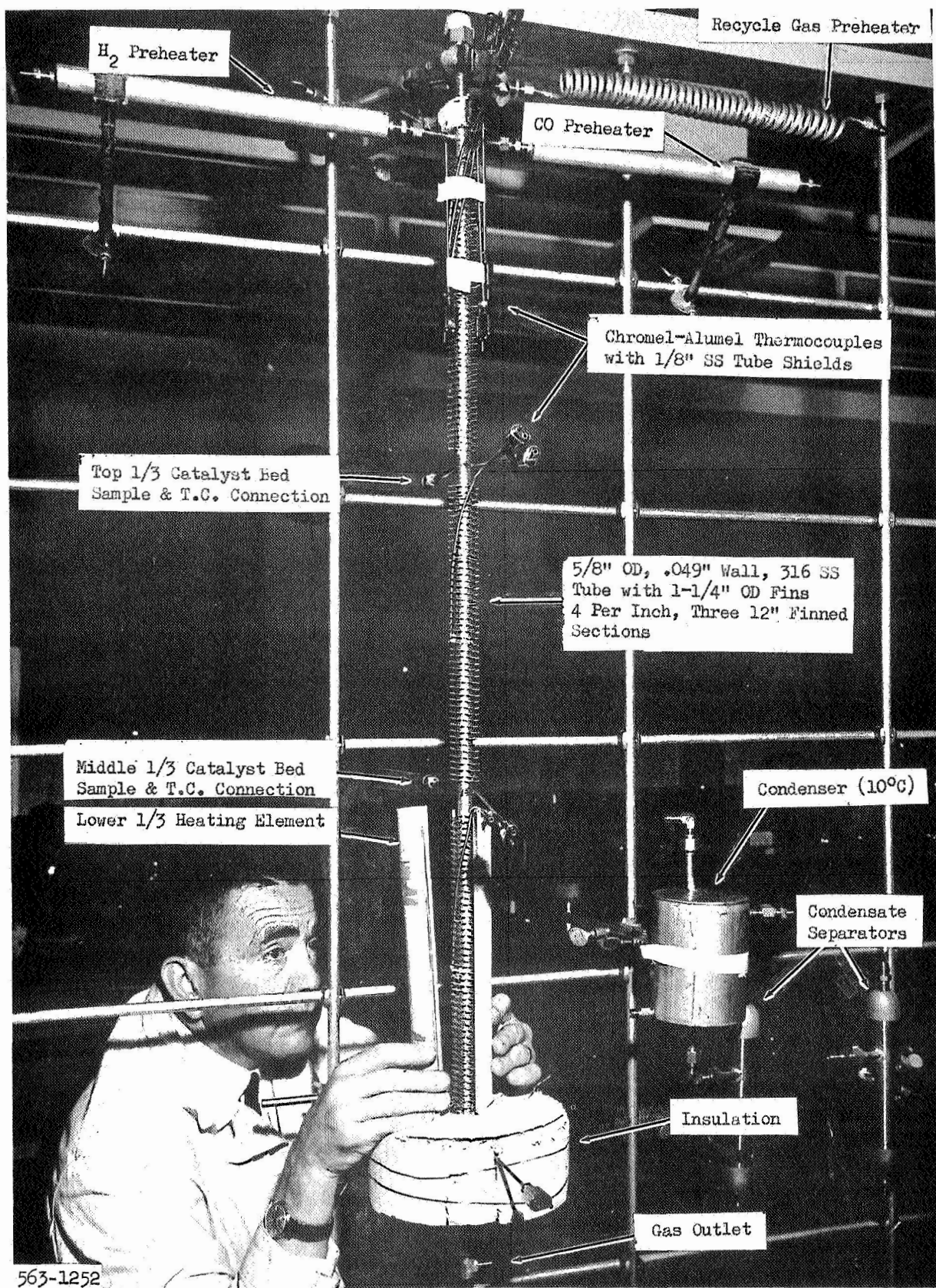
15 Sample Connection from Top 1/3 of bed

16 Sample Connection from Middle 1/3 of bed



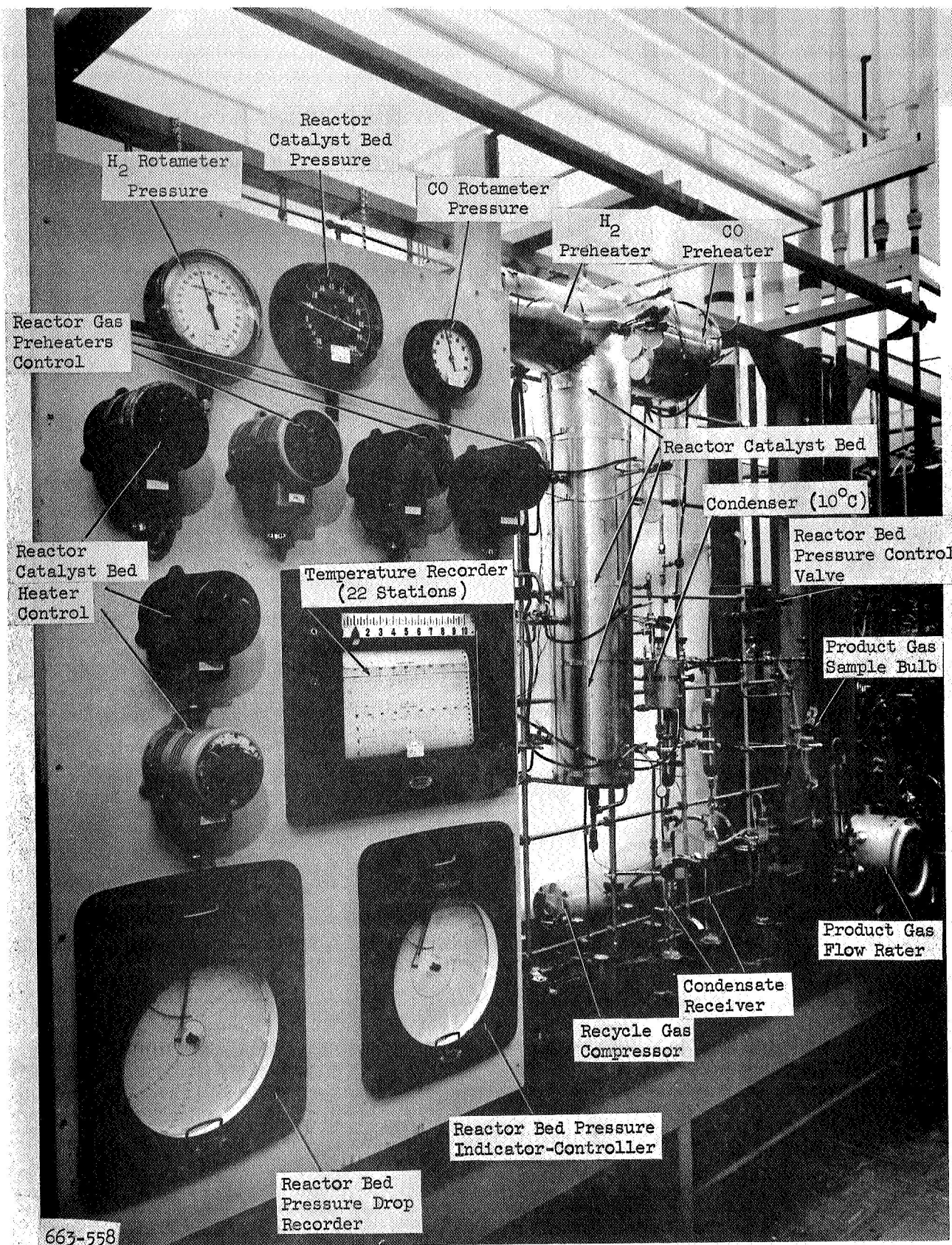
SCHEMATIC FLOW DIAGRAM OF H₂-CO REACTOR

Figure 1

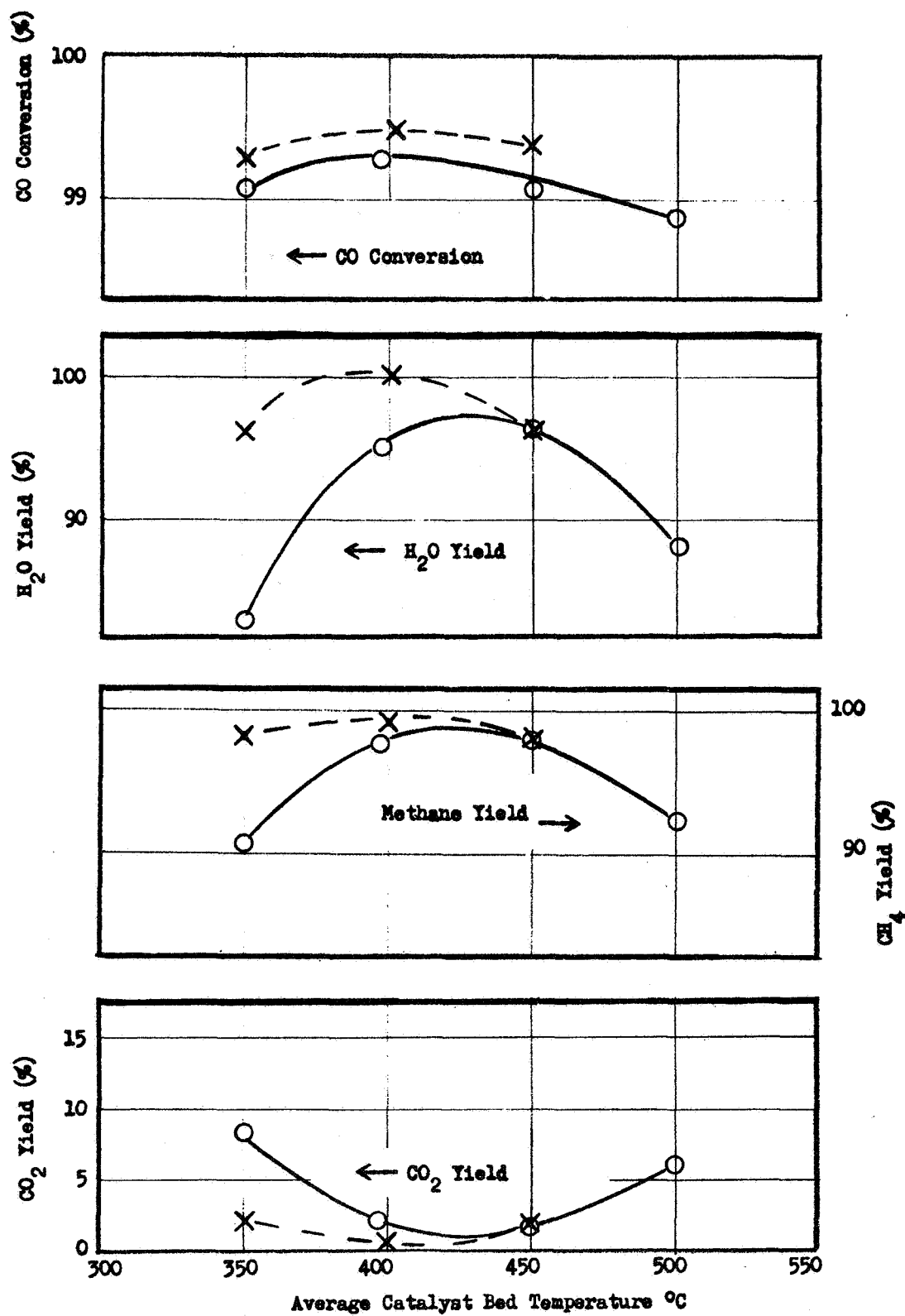


Flow Reactor for Reduction of CO with H₂ During Construction

Figure 2

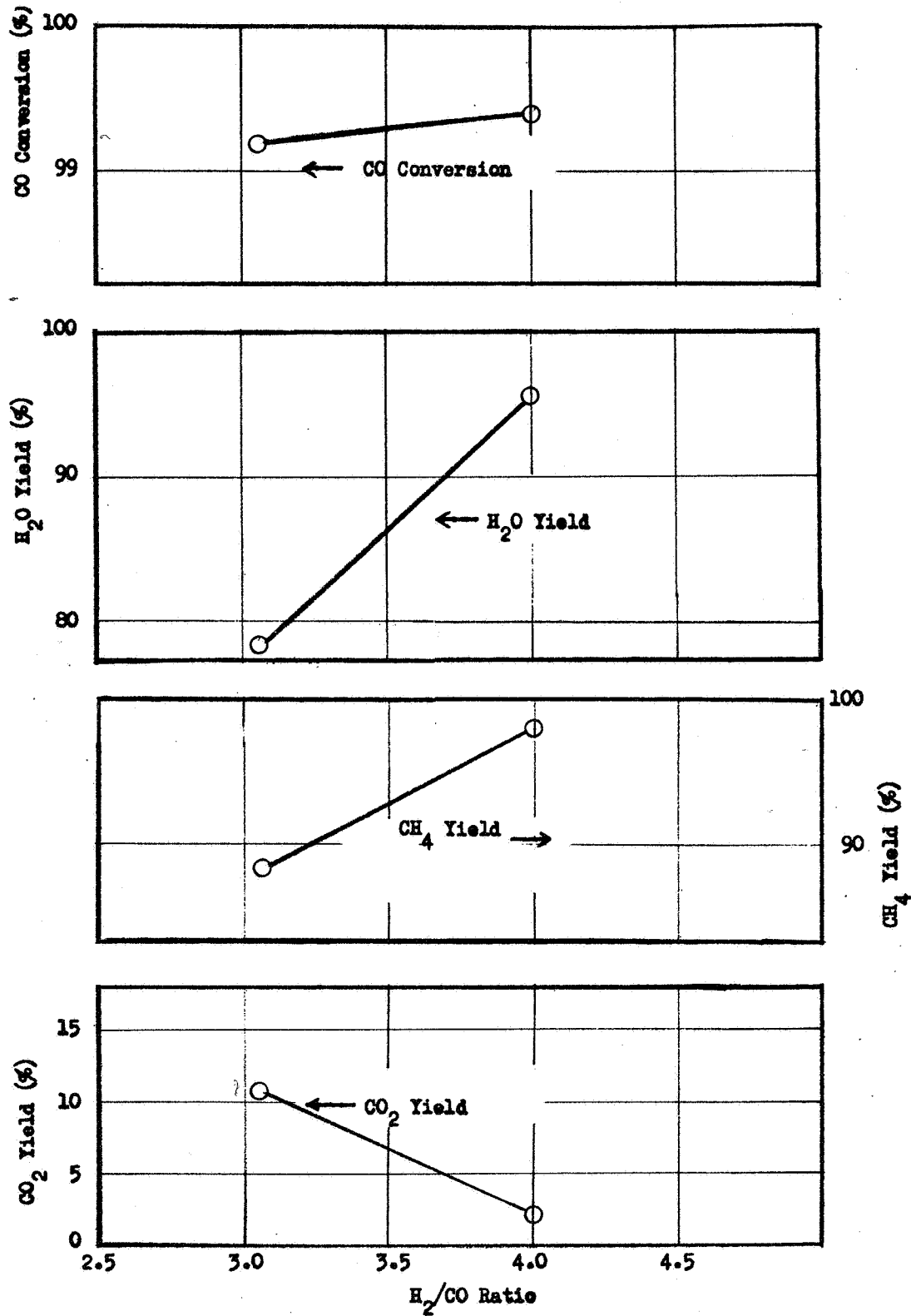


Flow Reactor for Reduction of CO with H₂

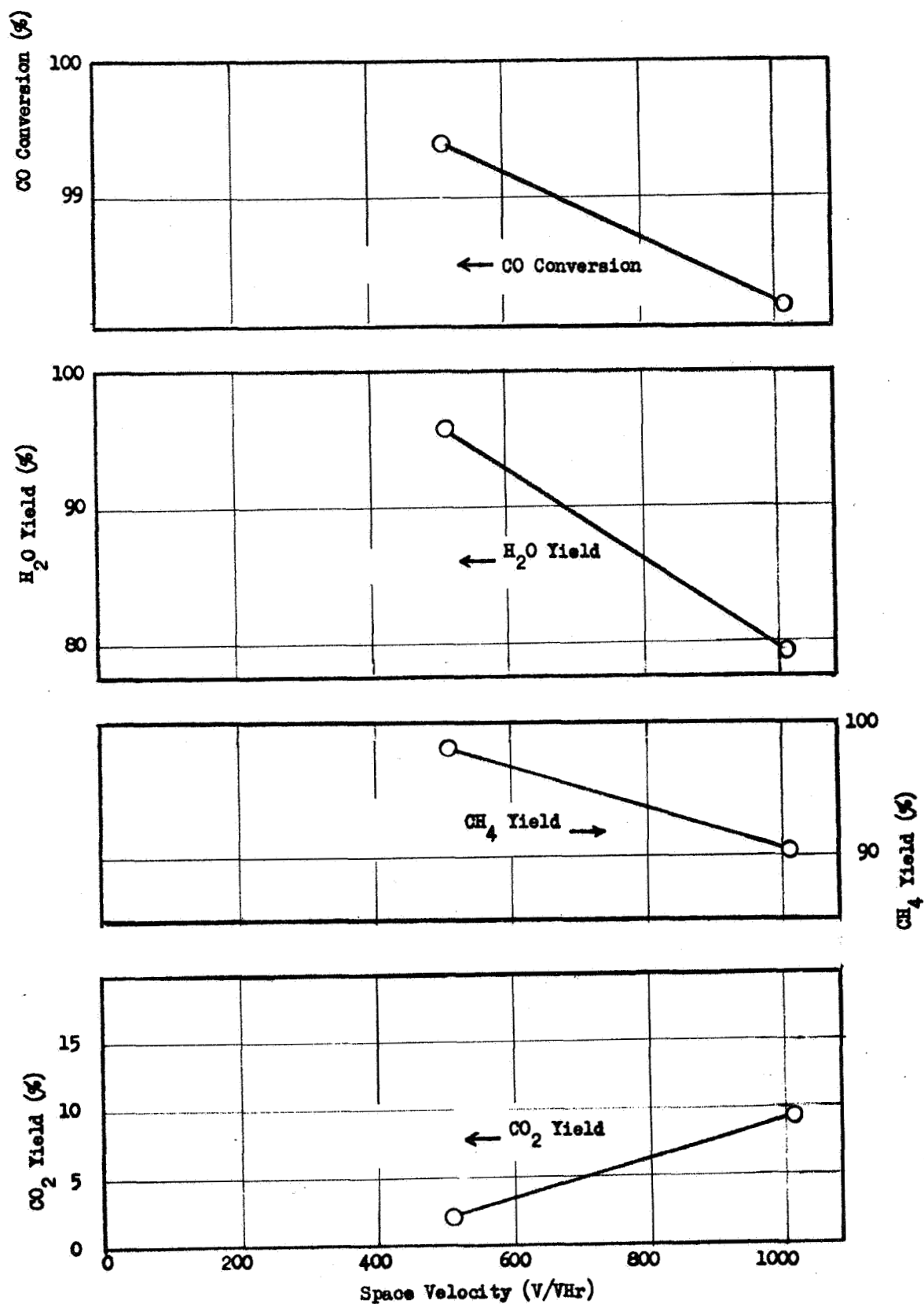


CO CONVERSION AND YIELDS VS TEMPERATURE
 4:1 H₂:CO Ratio; Atmospheric Pressure; 500 Space Velocity
 First Catalyst Charge ○ Second Catalyst Charge ×

Figure 4



CO CONVERSION AND YIELDS VS H_2/CO RATIO
500 SPACE VELOCITY; ATMOSPHERIC PRESSURE, 450°C



CO CONVERSION AND YIELDS VS SPACE VELOCITY
4:1 H₂/CO Ratio; Atmospheric Pressure; 450°C

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